L 14169-66 EWP(j)/EWT(m)/ETC(m)-6/T/EWP(v)RM/WW ACC NR: AP6003940 SOURCE CODE: UR/0374/ 5/000/005/0058/0065 Trostyanskaya, Ye. B. (Moscow); Poymanov, A. M. (Moscow); Babayevskiy, P., G. (Hoscow) ORG: none TITLE: Causes of strength difference in plastics with mineral powder fillers SOURCE: Mekhanika polimerov, no. 5, 1965, 58-65 TOPIC TAGS: phanolic plants, phenolformaldehyde, silicon plastic, fiberglass, resin, filler, hardowing, plastic strength, shermoclasticity minural, the destricts.

ABSTRACT: The effect of mineral powders used as fillers on the rate 41 and depth of the hardening of phenolformaldehyde and silicone resins has been investigated. It has been shown that the degree of this effect depends on the composition of the powder filler and on the method of its preliminary preparation. The fillers usually are the inhibitors or the catalysts of the hardening process. The fillers catalyzing the hardening process increase the strength and thermostability of plastics. fillers, introduced in small quantities into resins and used as binders for fiberglass plastics, level the speed and depth of hardening and increase the strength and the heat stability of plastics. Orig. art. has: Card 1/2 UDC: 678:539.4.019

| 3 figures | and 5 tables. | [Based | on author | 's abstrac | tl. |          | 0   |
|-----------|---------------|--------|-----------|------------|-----|----------|-----|
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PROSTYANSKAYA, Ye.B.: NEFEDOVA, G.Z.

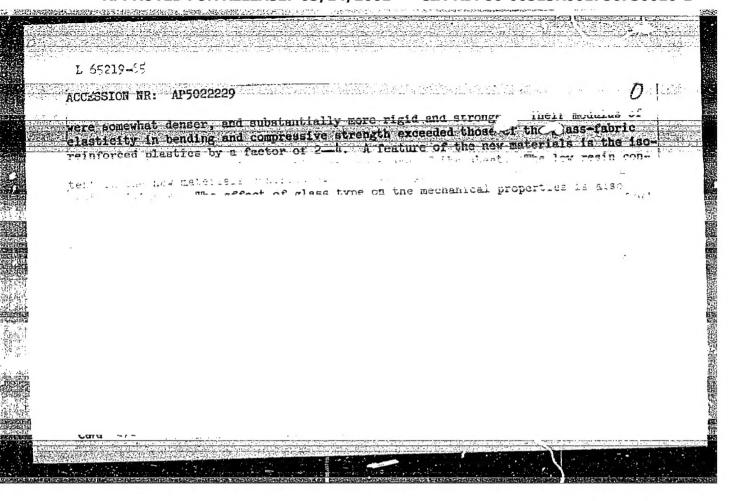
Insoluble polyelectrolytes with mobile functional groups. Vysokom, soed. 7 no.10:1767-1770 0 165.

1. Institut chistykh reaktivov i osobe chistykh khimicheskikh

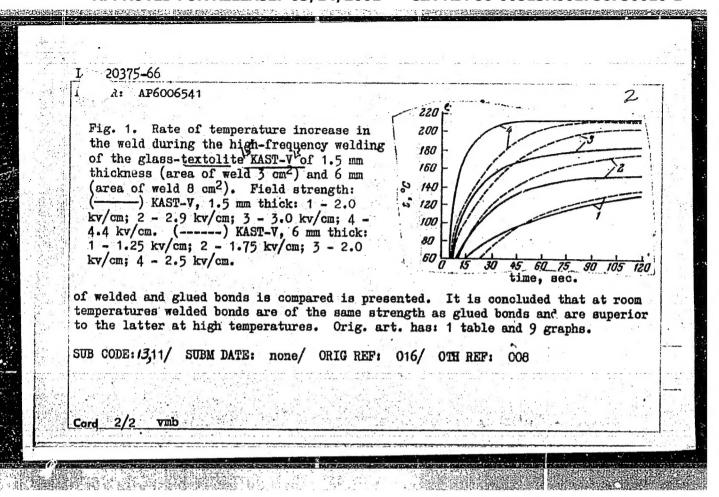
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| AUTHOR VALLEY  | yd1 <sup>b</sup>   | 8.744,3.01:539.4   | :65.085.5  |                    |
| AUTHOR: Vinogradov, V. M.; Never   | ov, A. N.; Bocharnikov   | . V. K. Troster  | n.420  |                    |
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| SCURCE: Flasticneskiye massy, no   | £ 1355 no  | 1V   |  |                    |
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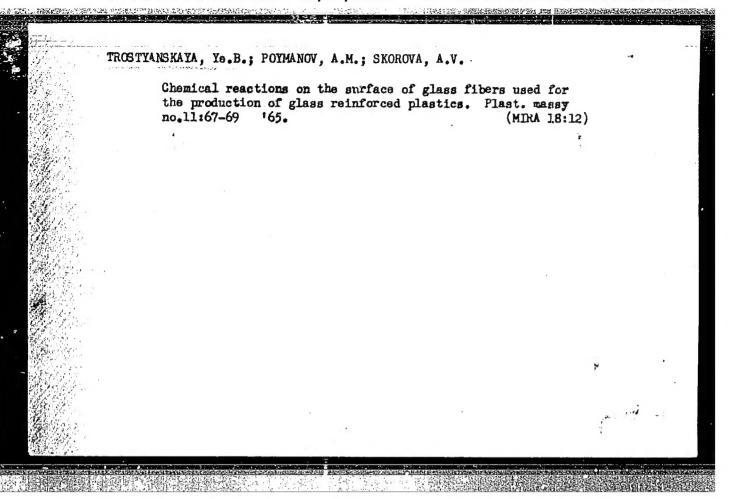
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| Terms Clearness of the print of the  | E a care can also                              |        |
| SOURCE: Plasticheskiyo massy, a  | . w. 1 . 1                                     |        |
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| the solvent from the "prepreg," the is sure did not exceed 10 kg/cm2. The or properties of the end products in tabu conventional glass-fabric reinforced p   | y-up was carried out in molds. Molding pres-   |        |

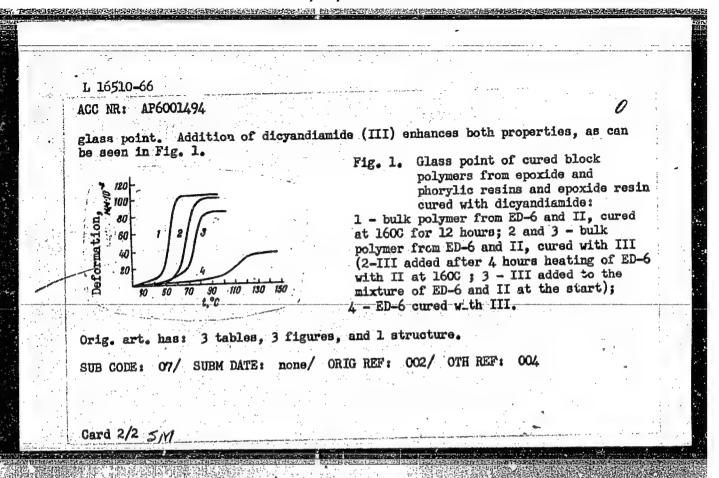


| 1100 1111  | AP6006541 JD/WW/ (A)   | SOURCE CODE:                                     | UR/0191/65/000/011/00                   | 22/002 |
|--|--|--|---|--------|
| AUTHORS:   | Trostyanskaya, Ye. B.;   | Komarov, G. V.; Shishk                           |   | 60     |
| ORG: nor   |  |  |   | B      |
| TITLE: J   | oining of hardened plast   | ics by the method of cl                          | nemical we! ng                          | D      |
| Carlos Company of the | Plasticheskiye massy, no   |  | , |        |
| TOPIC TAG<br>evalpation  | S: polymer, pastic, ulon, adhesive bonding   | trasonic welding, weldi                          | ing technology, weld                    |        |
| welding w  | The object of the investigation that the optimum condition as accomplished by a high | of requerer (1.1 V-1.                            | g of hardened plastic                   | s. Th  |
| type UZG-  | 10. Microsections of the   | tation of type UZP-1(t)                          | itilizing a generator                   | of     |
| nicrophot  | ography. The weld strang   | th of the warders 1                              | were investigated by                    |        |
|  |  | d during welding was m<br>(see Fig. 1), and a ta |   |        |





EWT(m)/EWP(v)/EWP(j)/T/ETC(m)-6 WW/RM SOURCE CODE: UR/0191/65/000/012/0012/0014 AP6001494 ACC NR: Trostyanskaya, Ye. B.; Venkova, Ye. S.; Pavlova, A. P. AUTHORS: ORG: none TITLE: Structural plasticization of epoxide resins SOURCE: Plasticheskiye massy, no. 12, 1965, 12-14 TOPIC TAGS: epoxy plastic, plasticizer, copolymer, adhesive / ED-6 epoxide resin ABSTRACT: This article deals with the preparation of phorylic resins (molecular weight of 400-600) by polycondensation of monophenylphosphate dichloroanhydride with resorcinol (I), hydroquinone (II), p,p'-dihydroxydiphenylmethane, and p,p'-dihydroxydiphenylisopropane according to a method described earlier (U. S. Pat. 2616873; Brit. Pat. 679834). Preparation and properties of bulk copolymer obtained by addition of I or II to epoxide resin ED-6 are also described. The work was undertaken in order to improve the impact strength and adhesive properties of polyepoxides. It was established that phorylic resins in bulk polymers function as structural plasticizers, considerably increasing impact and tensile strength of cast products and of adhesive films, at the same time lowering their UDC: 678.64414215:678.049 Card 1/2



TROSTYANSKAYA, Ye.B.; NEFEDOVA, C.Z.

Innoluble polycomplexons. Zhur. anal. khim. 20 no.8:775-780 (MERA 18:10)

1. Vsesoyuznyy nauchno-issledovateliskiy inshibut khimicheskikh reaktivov i osobo chistykh khimicheskikh veshchesty, Moskva.

| ・ 一   | Antieck                                    | , G. V.; Shishk   | 111, 10 110                                | 0         |
|---|--|---|--|-----------|
| ORG: none TITLE: Bonding cured glass-re   | einforced pla                              | astics. Class   | 39, No. 176388                             | 2         |
| SOURCE: Syulleten' izobreten  | iy i toverny?                              | kh znakov, no.  | 22, 1965, 56                               |           |
| TOPIC TAGS: glass, reinforced   |  |   |  |           |
| <br>ABSTRACT: An Author Certific forced plastics based on unsa by additional polymerization.  | turated poly<br>The method in              | nvolves coating   | of the surfaces to<br>erization initiator  | be bonded |
| ABSTRACT: An Author Certific forced plastics based on unsa by additional polymerization. With a monomer (e.g., styrene of the surfaces, and high-fre  | The method in a solution we equency or ul- | nvolves coating<br>with added polym<br>trasonic heating | of the surfaces to<br>perization initiator | be bonded |
| ABSTRACT: An Author Certific forced plastics based on unsa by additional polymerization. With a monomer (e.g., styrene of the surfaces, and high-free | The method in a solution we equency or ul- | nvolves coating   | of the surfaces to<br>perization initiator | be bonded |
| ABSTRACT: An Author Certific forced plastics based on unsa by additional polymerization. With a monomer (e.g., styrene of the surfaces, and high-fre  | The method in a solution we equency or ul- | nvolves coating<br>with added polym<br>trasonic heating | of the surfaces to<br>perization initiator | be bonded |

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| ACC NR: AP5026525  |   | UR/0286/65/000/019/0069/0069 |
| AUTHORS: Trostyans | પૂર્ષ<br>kaya, Ye. B.; Shishkin, V. A.; Golovi  | kin, G. S.; Komarov, G. V.   |
| ORG: none          |   | 28                           |
| TITLE: A method fo | r vulcanizing. Class 39, No. 175221 15  |                              |
|                    | isobreteniy i tovarnykh snakov, no. I   |                              |
|                    | ization, rubber, monomer, initiator   |                              |
| ABSTRACT: This Aut | thor Certificate presents a method for the method stability of the union, the surface of a proper monomer and an initiates are then brought into contact with o | or or with a vulcanizing     |
| SUB CODE: IE/      | SUBM DATE: 05May64  |                              |
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L 20382-66 EWP(a)/EWI(m)/EWA(d)/EWP(j)/T/EIC(m)-6 WW/RM/WH

ACC NR: AP6006550

(A).

SOURCE CODE: UR/0191/65/000/011/0067/0069

AUTHORS: Trostyanskaya, Ye. B.; Poymanov, A. M.; Skorova, A. V.

92

ORG: none

TITLE: Chemical reactions on the surface of glass fibers used in the manufacture of fiber-glass plastics/

SOURCE: Plasticheskiye massy, no. 11, 1965, 67-69

TOPIC TAGS: fiber glass, polymer, epoxy plastic, silicon quartz, surface active agent, glass fiber, silicon, glass, quartz, chemical reaction, surface ionization ABSTRACT: It was the object of this investigation to find suitable conditions for the modification of surfaces of glass, silicon, and quartz fibers. The modification was carried out by the chemical addition of organic radicals to the surface, according to the schemes

$$\equiv$$
SI-OH + SOCI<sub>3</sub>  $\longrightarrow \equiv$ SiCI + SO<sub>3</sub> + HCI  
 $\equiv$ SI-CI + BrMgR  $\longrightarrow \equiv$ SI-R + MgBrCI

and

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| ACC | NR:   | AP6006550 |               | o       |          |                 |
| J.  |       |           | =Si-CI + K-O- | _CC=CH2 | • ≡SiOC- | -C=CH2 - KCI    |
|     |       |           | - An-         | CH²     |          | CH <sub>3</sub> |

where R = trimethylsilane, allyl, ethyl, phenyl, or methacryl respectively. The contact angle of whetting and electrical surface conductivity of the modified surfaces were determined after the method of Ye. B. Trostyanskeya, A. M. Poymanov, and Yu. N. Kazanskiy (Plast. massy, No. 7, 1964). The experimental results are tabulated. It was found that substitution of mono- and divalent ions by tri- and tetravalent ions on the modified surfaces enhances the hydrophobic nature of the latter and eliminates the retarding action of the surface hydroxyl groups on the rate of hardening of polycondensation type binders, thus improving the qualities of the fiber-glass plastics. Orig. art. has: 3 tables and 4 equations.

SUB CODE: 11/ SUBM DATE: none/ ORIG REF: 008/ OTH REF: 007

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#### CIA-RDP86-00513R001756730010-1

L 20406-66 ENT(m)/EMP(j)/T/ETC(m)-6 WW/RH

ACC NR: AP6008402

(A)

SOURCE CODE: UR/0374/66/000/001/0067/0073

AUTHOR: Trostyanskaya, Ye. B.; Novikov, V. U.; Kazanskiy, Yu. N.

48

ORG: Moscow Aviation Technological Institute (Moskovskiy aviatsionno-tekhnologicheskiy institut)

TITLE: Effect of increased temperatures on the strength of solidified resins and of materials of the same base. 1. Effect of increased temperatures on the strength of solidified phenolformaldehyde resins

SOURCE: Mekhanika polimerov, no. 1, 1966, 67-73

TOPIC TAGS: resin, phenolformaldehyde, temperature dependence, tensile strength, compressive strength, thermal effect

ABSTRACT: An investigation of changes in tensile strength and of weight diminution in phenolformaldehyde resins was carried out under high temperature conditions. It was revealed that a spontaneous transition from the first to the second and third structural stages takes place with concomitant increase in the stabilization of strength properties in the process of thermal destruction. In all the resins investigated and for every structural stage, the direct dependence between the relative change of ultimate compression strength and the relative change of weight were established irrespective of the conditions of thermal treatment. The investigation was carried out on standard samples obtained by molding a mixture of hardened resin and powder of the same, but preliminarily hardened, resin. Samples produced in this Cord 1/2

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| made of mole<br>the process<br>furfural-for | tter physical a<br>ling powders wi<br>of transition<br>rmaldehyde resi<br>olic-aniline-fo<br>uthors' abstrac | from one stains, and the ormaldehyde | ige to anoth | er is typical | of the phenol | ic-<br>s typical |
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### CIA-RDP86-00513R001756730010-1

UR/0190/66/008/007/1219/1225 ENT(m)/ENF(j)/T SOURCE CODE: 35857-66 ACC NR: AP6023429 111

AUTHOR: Trostyanskaya, Ye. B.; Venkova, Ye. S.; Aristovskaya, L. V.

ORG: Moscow Aviation Technology Institute (Moskovskiy aviatsionnyy tekhnologicheskiy

TITLE: Polycondensation of tris(hydroxymethyl)phosphine oxide with phenols institut)

SOURCE: Vysokomolekulyarnyye soyedineniya, v. 8, no. 7, 1966, 1219-1225

TOPIC TAGS: organic phosphorus compound, alkylphosphine oxide, polymerization rate, polycondensation, catalytic polymerization, heat resistant plastic, thermosetting material, phenolic plastic

ABSTRACT: Controversial data on the reaction of tris(hydroxymethyl)phosphine oxide (TMPO) with phenol prompted a study of the reactions of TMPO with phenol, resorcinol, and phloroglucinol. The purpose of the study was to prepare phosphorus containing and phiorogracinol. The purpose of the study was to prepare phosphorus containing polymers with increased heat and strength resistance in the absence of a catalyst, the reaction with phenol at 160—200C yielded only a phenol-formaldehyde type resin with a low polycondensation rate because of predominant splitting of TMPO. In the presence of a BF3-phenol complex as catalyst 1 the rate of polycondensation was increased and at 1450 in 8 hr the reaction yielded 30 wt% of an insoluble, thermosetting phenol-TMPO resin with high phosphorus content. The polycondensation rate was further increased in the reaction with resorcinol and even more so with phloroglucinol. Chemical analysis of the reaction products at various stages of polycondensation indicated Card 1/2

#### L 35857-66

, ACC NR: AP6023129

a nearly total polycondensation of TMPO with resorcinol and total with phloroglucinol. ·Final polycondensation products with both resorcinol and phloroglucinol were insoluble thermosetting resins with nearly theoretical phosphorus content (10.9-11.4% P). Physicomechanical characteristics (Table 1) of the synthesized and hardened P-containing polymers are comparable to those of the phenol-formaldehyde resins, but their heat resistance is significantly higher. A sharp increase in deformation was observed

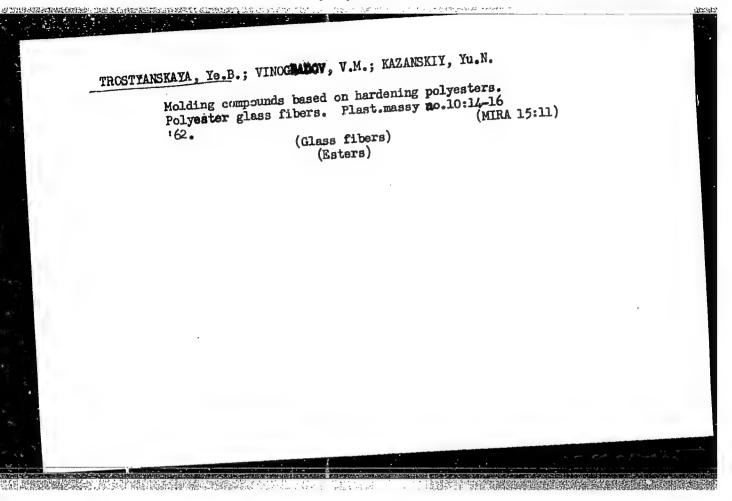
Table 1. Properties of compacted TMPO-resorcinol

| 100            |                             | -                                     |       |                               |                   |                  |                  |                  |
|----------------|-----------------------------|---------------------------------------|-------|-------------------------------|-------------------|------------------|------------------|------------------|
| :              | E S                         | ti .                                  | at at | hr.                           | Heat re           | sistance         | at 9000 1        | or 5 min         |
| Resin<br>type  | Compressio<br>strength, lay | Specific imper<br>foughness,<br>kg/cm | Bet   | Water sbscr<br>taion, % in 24 | Ignition,<br>sec. | Burning<br>time, | Flame -          | Coke<br>residue, |
| TMP0-resorcino | 970                         | 5,55                                  | 4,46  | 0,8                           | 7-8               | 20-25            | Webdy<br>smoking | 52,6             |
| mwpo-phloro-   |                             | 5,62                                  | 4,68  | 0,19                          | 15-18             | 10-15            | Light-<br>bluish | 59,1             |
| glucinol       | ,                           |                                       | 1     | * - 1                         | 4 18              |                  |                  | -                |

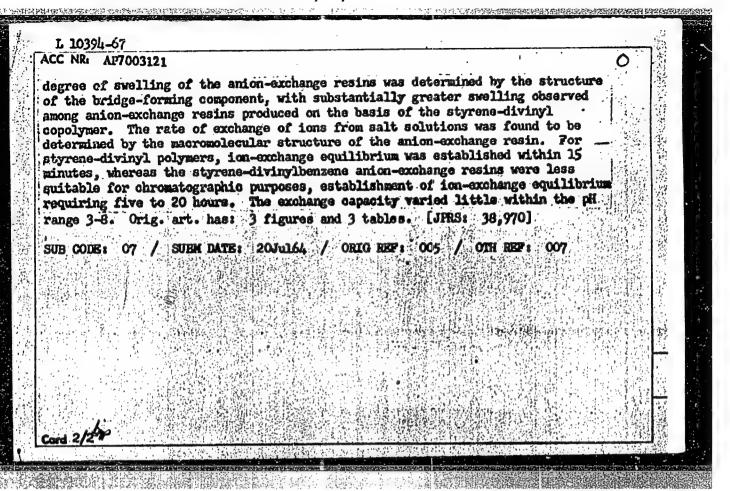
at 260-270C for TMPO-resorcinol resin and at 320-350C for TMPO-phloroglucinol resin. Orig. art. has: 6 tables:

SUB CODE: 07/ SUBM DATE: 11Jun65/ ORIG REF: 006/ ORIG REF: ATD PRESS:

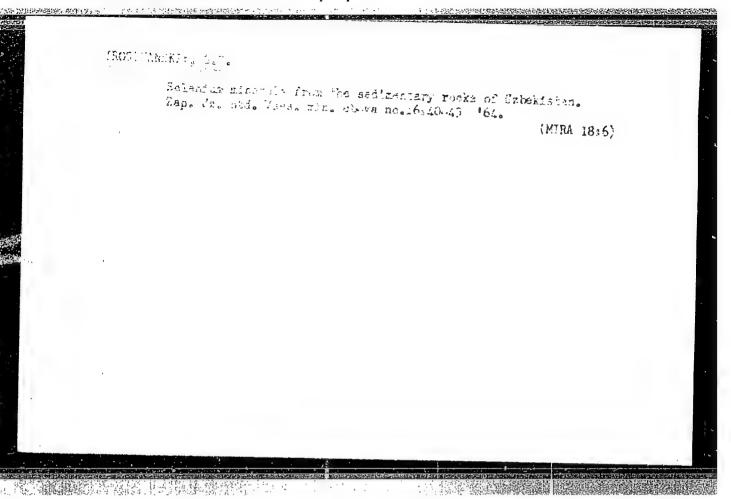
Card 2/2 /20



SOURCE CODE: UR/0080/66/039/008/1754/1760 DS/RM L 10394-67 EWT(m) ACC IRI AP7003121 AUTHOR: Trostyanskaya, Ya. B.; Makarova, S. B. ORG: All-Union Scientific Research Institute of Chemical Reagents and Especially Pure Chemical Substances (Vsesoyuznyy nauchno-issledovatel'skly inst) tut 生する khimicheskikh reaktivov i osobo chistykh khimicheskikh veshchestv) TITIE: Anion-exchange resins belonging to the class of onium compounds SOURCE: Zhurnal prikladnoy khimii, v. 39, no. 8, 1966, 1754-1760 TOPIC TAGS: anion exchange resin, chromatography, copolymer, styrene, vinyl compound ABSTRACT: Chromatographic separation of ions from salt solutions requires that the ion-exchange resin possess multifunctionality, a high degree of ionization in a broad range of pH values, and a high rate of establishment of equilibrium in the exchange reaction with ions of the solution. Copolymers of styrene with divinylbenzene and styrene with divinyl with various amounts of the bridgeforming component in the copolymer, were used to synthesize anion-exchange resins for a study of the influence of structure of the macromolecules upon the ion-exchange properties of the resins. The copolymers were chloromethylated, and then the chlorine atom replaced by amines, phosphines, or sulfides. Anionexchange resins with sulfonium and phosphonium structural groups were readily decomposed in solutions of alkal' anion-exchange resins tested with ammonium functional groups acquired the structure of bases, the degree of association of which was determined by the structure of the radicals on the quaternary nitrogen. The thermal stability of the anion-exchange resins in the salt form decreased in the series ammonium > phosphonium > sulfonium compounds. 1935 Card 1/2



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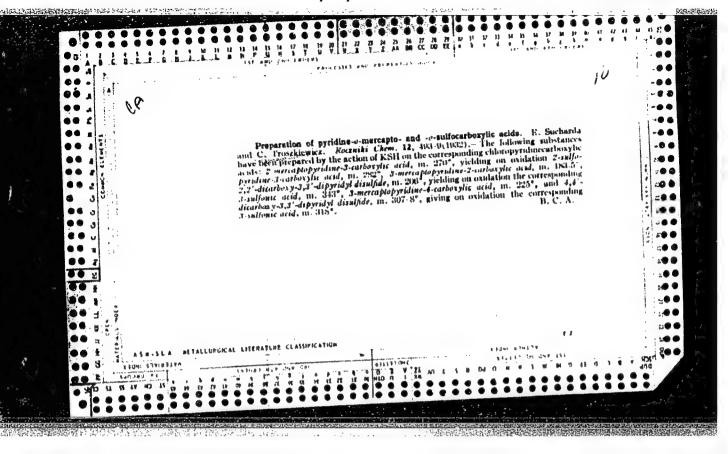
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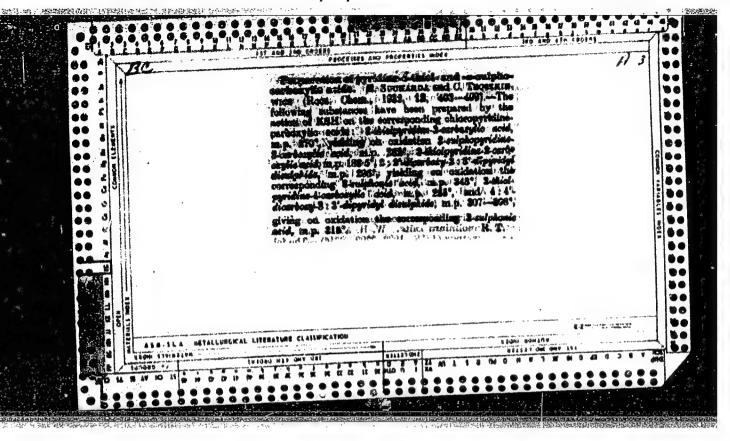
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TROSZKIEWICZ, Czeslaw; GOSZCZYNSKI, Stefan

Cyclization of benzylideneacetone and  $\beta$ -methylbenzylideneacetone oximes to the quinoline nucleus. Rocz chemii 37 no. 7/8:919-920 163.

1. Department of Organic Chemistry, Institute of Technology, Gliwice.

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GLINKA, Jadwiga; TROSZKIEWICZ, Czeslawa

Synthesis of quinoline nucleus by cyclization of oximes. Rocz chemii 37 no.12:1643-1644 163.

1. Department of Organic Chemistry, Technical University, Gdansk.

Alkylation of aromatic compounds with substituted amides RNHCOR' and other reactions of amides in the presence of PCCl<sub>3</sub>. Rocz chemii 36 no.5:853-864, '62.

1. Department of Organic Chemistry, Technical University, Cliwice.

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Structure of the benzyl group structure of the amide Archrnhcocoh5 and its influence upon the course of the bensylation reaction of aromatic compounds. Rocz chemii 36 no.5:843-851 62.

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Influence of the structure of the remaining acyl group in C6H5CH2NHCOR amide in the course of benzylation of aromatic compounds. Rocz chemii 36 no.2:265-274 62.

1. Department of Organic Chemistry, Silesian Institute of Technology, Gliwice.

8/081/62/000/021/016/069 B156/B101

AUTHORS: Prajenar, Bronisław, Troszkiewicz, Czesława

TITLE: Relationship between the properties of alkylating N-benzylamides and the nature of the condensing agent

PERIODICAL: Referativnyy zhurnal. Khimiya, no. 21, 1962, 148, abstract 21Zh108 (Roczn. chem., v. 35, no. 6, 1961, 1635 - 1639 [Pol.; summaries in Russ. and Eng.]).

TEXT: When m-xylene (I) is benzylized by the action of  $C_6H_5CH_2NHCOC_6H_5$  (II) in the presence of dehydrating and condensing substances (DCS)  $[P_2O_5\ (III),\ POCl_3\ (IV),\ PCl_5,\ PBr_5,\ PCl_3,\ SOCl_2\ or\ AlCl_3],\ products of mono- and dibenzylizing are only obtained when using III or IV. The by-products are <math>C_6H_5CH_2Cl$  (V),  $C_6H_5CN$  (VI) and HCl, their formation being possible by the following scheme: II +  $PCl_5 \longrightarrow C_6H_5CH_2N=CClC_6H_5$  (VII) +  $POCl_3$ ; when heated, VII decomposes to form V and VI; VIII +  $H_2O \longrightarrow II+HCl$ . I,  $C_6H_5CH_3$  (VIII) and anisole (IX) have been benzylized by the action of Card 1/3

Relationship between the ...

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C6H5CH2NHCOCH3 (X) in the presence of IV. According to Schotten-Baumann, 30 g  $C_{6}H_{5}CH_{2}NH_{2}$  (XI) and 55 g  $C_{6}H_{5}COC1$  yield 95.3 % II, m.p. 106 - 107°C. After 25 g XI and 30 g C6H5COOH have been heated at 160 - 180°C for 6 hrs, the total yield of II from the melt is 79.1 %. 0.1 moles II, 100 ml I, and a corresponding amount of DCS are heated for 3 hrs at 120°C (reflux condenser; the P205 is added in two batches: 35 g at the start, and a further 20 g 30 min later); when cool, it is dissociated with ice, and after a few hours 20 ml I are drawn off with the water layer, and the reaction products separated from the combined organic layers. The DCS are given, also the benzylizing products of I, the other reaction products, and the amount of recovered II in g: 55 g III, 57.3 % monobenzy1-I (XII) and 9.8 % dibenzy1-I (XIII), 5.7 g VI, -; 37 g IV, 71.5 % XII and 9.8 % XIII, 164 g V, 9.2 g VI and HCl, -; 42 g PCl<sub>5</sub>, -, 5.76gVV, 1.59 g VI and HCl, 11.5; 90 g PBr<sub>5</sub>, -, 44 g of a liquid substance with a boiling point of 70 - 90°C/16 mm Hg con-(content of VI 6.4 g, content of Br 34.7 %) and HC1, 4.2; 50 g PCl3, -, 0.15 g V, 0.1 g VI and HCl, 14; 71 g SOCl<sub>2</sub>, -, 1.32 g V, 1.16 g VI and HCl,

Relationship between the...

S/081/62/000/021/016/069 B156/B101

13.2; 42 g AlCl<sub>3</sub> (in toluene), solid unidentified product, -. For 3 hrs 25 g X, 66 g IV and 150 ml of I, VIII or IX are heated at 120°C; they are dissociated with ice, and the products of the reaction separated (the initial substance and the reaction products are given): VIII, 45.6 % benzyl toluene (b.p. 139 - 141°C/11 mm Hg,  $n_D^{20}$  1.5715), 7.05 % of dimbenzyl toluene (b.p. 215 - 235°C/11 mm Hg,  $n_D^{20}$  1.5955) and 11.8% V; I, 46.1 % XII (b.p. 157 - 158°C/14 mm Hg,  $n_D^{20}$  1.5697), 3 % XIII (b.p. 235 - 245°C/12 mm Hg,  $n_D^{20}$  1.5972) and 9.4% V, b.p. 60 - 70°C/11 mm Hg; III, 57.7 % benzyl anisole (b.p. 157 - 160°C/10 mm Hg,  $n_D^{20}$  1.5773) and 8.3 % dibenzyl anisole, b.p. 200 - 210°C/2 mm Hg,  $n_D^{20}$  1.6010. [Abstracter's note: Complete translation.]

Card 3/3

5/081/62/000/023/034/120 B166/B101

AUTHORS:

Prajsnar, Bronisław, Troszkiewicz, Czesława

TITLE:

The effect of the structure of the acyl residue of

C6H5CH2NHCOR amide on the course of the reaction of aromatic

compound benzylation

PERIODICAL:

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Referativnyy zhurnal. Khimiya, no. 23, 1962, 257, abstract 23Zh137 (Roczn. chem., v. 36, no. 2, 1962, 265-274 [Pol.;

summaries in Russ. and Eng. ])

TEXT: A study has been made of the reaction of toluene benzylation by the action of  $C_6H_5CH_2NHCOR$  (Ia-k, where (a) R = H, (b) R =  $CH_3$ , (c) R =  $C_2H_5$ ,

(d)  $R = tert - c_4^{H_9}$ , (e)  $R = cH_2^{C1}$ , (f)  $R = cGl_3$ , (g)  $R = c_6^{H_5}$ , (h)  $R = o - NO_2^{C_6}$ 

(i)  $R = p-NO_2C_6H_4$ , (k)  $R = p-CH_3OC_6H_4$ ); also by the action of N-benzyl benzene sulfonamide (II) and of N-benzyl phthalimide (III) in the presence of POCl3. The dependence of benzyl toluene (IV) yield on the structure of the acyl residue of I-III is used to show that the reaction mechanism Card 1/4

The effect of the structure of ...

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is one of electrophilic substitution. A similar reaction of Ig with  $C_6^H_6$ , toluene, m-xylene, anisole and  $C_6^H_5^Br$  gave their mono- and dibenzyl substitutes. The reaction between Ig and  $C_6^H_5^{NO}_2$  does not go. The method of producing Ia, c, d, e has been improved and Ih was synthesized. A mixture of 25 g  $C_6^H_5^{CH}_2^{CH}_2^{NH}_2$  (V) and 50 ml 90% HCOOH is boiled for 30 min, the excess acid is removed by vacuum distillation, the product is Ia, yield 28.5 g, b.p. 169-1720/11 mm Hg, m.p. 63-64°C (from gasoline-benzene 4:1). A mixture of 25 g V and 70 g  $C_2^H_5^{COOH}_5^{C$ 

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yield 40.2 g, m.p. 93-94°C (from alcohol). A mixture of 42 g o-NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>COOH and 45 g SOCl<sub>2</sub> in 150 ml C<sub>6</sub>H<sub>6</sub> is boiled for 1 hr, 80 ml of fluid is distilled off and a solution of 54 g V in 50 ml C<sub>6</sub>H<sub>6</sub> is added a drop at a time to the residue, this mixture is boiled for 20 min diluted with water and the product is Ih, C<sub>14</sub>H<sub>12</sub>O<sub>3</sub>N<sub>2</sub>, yield 72%, m.p. 124-125°C (from alcohol). A mixture of 0.1 g mole I, 100 ml toluene and 37 g POCl<sub>3</sub> is heated for 3 hrs at 120°C, cooled, ice is added, this is then filtered and IV is extracted with toluene (the I-III are given and the yield of IV as %): Ia, 1.65; Ib, 45.5; Ic, 25.2; Id, 48.7; Ie, 25.4; If, 0; Ig, 70.7; Ih, 60.5; Ii, 70; Ik, 61; II (P<sub>2</sub>O<sub>5</sub> instead of POCl<sub>3</sub>), 36; III, 0. A mixture of 0.1 mole Ig, excess aromatic hydrocarbon and 37 g POCl<sub>3</sub> is heated for 3 hrs at 140°C, cooled, ice is added and the appropriate mono- and dibenzyl derivatives are separated (the following table gives the aromatic hydrocarbon, quantity in g, the monoderivative produced, its yield %, boiling point in °C/mm Hg, n<sub>D</sub><sup>20</sup>, the di-derivative produced, its yield %,

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boiling point in  ${}^{O}C/mm$  Hg,  ${}^{O}D$ ):  ${}^{C}_{6}{}^{H}_{6}$ , 70, diphenyl methane, 38, 126-128.5/11, m.p. 26-27°C, -, dibenzyl benzene, 11.6, 218-224/12, dibenzyl toluene, 80, benzyl toluene, 70.7, 140-143/12, 1.5710, 71.5, 148-152/11, 1.5694, dibenzyl-m-xylene, 90, benzyl-m-xylene, anisole, 100, benzyl anisole, 78, 162-165/11, 1.5769, dibenzyl anisole, 11.1, 220-240/12, 1.6010; bromobenzene, 120, benzyl bromobenzene, 22.2, into p-C $_{6}{}^{H}_{5}$ CH $_{2}{}^{C}_{6}{}^{H}_{4}$ COOH, m.p. 158-159°C), -, -, -, -, [Abstracter's note:

Card 4/4

S/081/63/000/004/020/051 B187/B102

AUTHORS:

Prajenar, Bronisław, Troszkiewicz, Czasława

TITLE:

On the alkylation of aromatic compounds with substituted amides RNHCOR' and on other reactions of amides in the

presence of POCL

PERIODICAL:

Referativnyy zhurnal. Khimiya, no. 4, 1963, 242-243, abstract 4Zh132 (Roczn. chem., v. 36, no. 5, 1962, 853-864

Pol.; summaries in Russ. and Eng. )

TEXT: The following particular facts can be established about the reactions of the smides having the general formula RNHCOR' (I) with aromatic compounds (ARV) in the presence of  $POCl_5$ : (a) when  $R = (C_n H_{2n+1})_2$ -CH or  $R = (C_n H_{2n+1})_5$ C, R is easily broken off; this leads to the formation of alkylation products of the ARV, unsaturated hydrocarbons, other substitution products and corresponding nitriles. (b) when  $R = C_n H_{2n+1} CH_2$ , the C-N bond is considerably more stable and these amides are not subjected to deamination if they are influenced by  $POCl_3$ ; on the other hand, Card 1/7

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On the alkylation of ...

different reactions come about depending on the kind of acyl; e.g., in case of I (R =  $C_4H_9$ , R' =  $C_6H_5$ ) (Ia) deacylation comes about with the formation of  $C_6H_5$ COCl. The reactions, both the alkylation and the elimination, are apparently monomolecular with the usual stage of the carbonium ion formation K<sup>+</sup>. As regards orientation both reactions proceed according to the Zaitsev rule. Reducing 10 g of  $(CH_3)_2CHCH=NOH$  in 100 ml of alcohol with Na (50% excess) gives 40% ( $CH_3$ ) $_2CHCH_2NH_2$ , b.p.  $67-69^{\circ}C$ , which is benzoylated by the Schotten-Baumann method; extraction with ether and removal of the solvent yields I [R =  $(CH_3)_2CHCH_2$ , R' =  $C_6H_5$ ] (Ib), b.p. 170-171° C/11 mm, m.p.  $56.5-67^{\circ}C$ . 50 g of  $C_5H_1C(CH_3)$  = NOH (b.p. 98-99°C/11 mm), 55 g of Na and 56 ml of alcohol are used to synthesize 62%  $C_5H_1CH(NH_2)CH_3$ , b.p. 139-142°C in an analogous manner; I [R =  $C_5H_{11}CH(CH_3)$ , R' =  $C_6H_5$ ] (Ic), m.p. 70-71°C is obtained from this by the Schotten-Baumann method (from thinned alcohol).  $(CH_3)_3COH$  (II) is benzoylated up to I [R =  $(CH_3)_3C$ , R' =  $C_6H_5$ ], (Id). 19.3 g of Card 2/7

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concentrated H<sub>2</sub>SO<sub>4</sub> is added dropwise to 13 g of (CH<sub>3</sub>)<sub>3</sub>COH (II) and 19.2 g of C<sub>6</sub>H<sub>5</sub>CN (III) over a period of 15 minutes at a temperature of less than 40°; the mixture is heated at 40°C for one hour, 150 g of ice are added and then 84% Id, m.p. 133.5-134°C are obtained (from benzene). If concentrated H<sub>2</sub>SO<sub>4</sub> is added to a mixture of I and II in glacial CH<sub>3</sub>COOH at 50°C, the yield of Id is 72%. For other I obtained by various methods R, R', b.p. in °C/mm, m.p. in °C are given: cyclo-C<sub>6</sub>H<sub>11</sub>, CH<sub>3</sub> (Ie), 160-161/15, 104-104.5; cyclo-C<sub>6</sub>H<sub>11</sub>, C<sub>6</sub>H<sub>5</sub> (If), -, 149-150; Ia, 181-184/13, -; C<sub>2</sub>H<sub>5</sub>CH(CH<sub>3</sub>), C<sub>6</sub>H<sub>5</sub> (Ig), -, 85-86; C<sub>6</sub>H<sub>13</sub>, CH<sub>3</sub> (Ih), 141-142/10, -, n<sup>2</sup>O<sub>2</sub>D 1.4459; C<sub>6</sub>H<sub>13</sub>, C<sub>6</sub>H<sub>5</sub> (Ii), 212-215/23, 44-45; C<sub>7</sub>H<sub>15</sub>, CH<sub>3</sub> (Ik), 151-153/11, -, n<sup>2</sup>O<sub>2</sub>D 1.4468. Cyclo-C<sub>6</sub>H<sub>11</sub>NHSO<sub>2</sub>C<sub>6</sub>H<sub>5</sub> (IV), m.p. 90-91° was also obtained. C.1 moles of Id and 36 g of POCl<sub>3</sub> are heated 2.5 hours at 135° (bath temperature) in 90 ml of M-xylol (V) (separation of HCl), ice is added, and the non-reacted V, containing cyclohexene (VI) (yield 66%, determined by bromination), CH<sub>3</sub>CN and 3.8% cyclohexyl-M-xylol (VII), b.p. 130-140°C/13 mm, Card 3/7

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11.16 g of Br<sub>2</sub> are necessary to saturate the fraction containing V. This corresponds to a cyclohexene yield of 65.6%. For analogous reactions of I with ARV in the presence of POCl<sub>3</sub>, the initial substances, reaction conditions, reaction products (in all cases separation of HCl) are now given: 0.1 mole If and 36 g POCl<sub>3</sub> in 100 ml toluene, 120°C, 2.5 hours, 45% cyclohexane (VIII), 23% cyclohexyl toluene (b.p. 253-255°C, 136-138°C/23 mm, n<sup>18</sup>D 1.5240) and 9.1 g III; 0.1 mole If and 36 g POCl<sub>3</sub> in 100 ml V, 140°, 2.5 hours, 31.4% VIII, 35.6% VII (b.p. 135-139°C/12 mm, n<sup>18</sup>D 1.5253) and 8 g III; 0.1 mole If and 36 g POCl<sub>3</sub> in 100 ml anisole (IX), 140°C, 2.5 hours, 8.3% VIII, 67.8% cyclohexyl anisole (X) (b.p. 141-143°/11 mm, n<sup>18</sup>D 1.5330; on letting it stand for a while, 1.42 g p-X, m.p. 58-59°C settle; When the filtrate is cooled down to -10°, o-X is separated, m.p. 265-270°C, n<sup>20</sup>D 1.5305 together with 7.29 g III; 11.45 g Ic and 18.5 g POCl<sub>3</sub> in 50 ml IX, 3 hours, 140°C, 76% III, 53.5% (1-methyl hexyl-

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anisole (C<sub>14</sub>H<sub>22</sub>O, b.p. 133-136°C/11 mm, n<sup>20</sup>D 1.4970) and a certain amount of an unsaturated compound; 20.3 g If and 25 g POCl<sub>3</sub>, 1 hour, 150-160°C (bath temperature) with distillation of the reaction products, 73% VI (b.p. 81-82°C), a certain amount of III and chloro cyclohexane; 21.9 g Ic and 30 g POCl<sub>3</sub>, as in preceding case; 67.4% heptene-2 (b.p. 97-100°C, n<sup>20</sup>D 1.4043; oxidation with KMnO<sub>4</sub> leads to C<sub>4</sub>H<sub>9</sub>CCOH, b.p. 180-185°C; anilide, m.p. 60-61°) a certain amount of III and 0.3 g C<sub>5</sub>H<sub>11</sub>CHClCH<sub>3</sub> (b.p. 142-146°C, n<sup>20</sup>D 1.4273); 17.7 g Ig and 36 g POCl<sub>3</sub> in POCl<sub>3</sub> in 90 ml V, 3 hours, 120°C (bath temperature), 64.5% butene-2 (distilled off during the reaction and absorbed in a solution of Br<sub>2</sub> in CCl<sub>4</sub>); (CH<sub>3</sub>CHBr)<sub>2</sub>, b.p. 154.5-157°C, n<sup>20</sup>D 1.5104), 82% III and 6.8% sec-butyl-M-xylene (b.p. 190-200°, n<sup>20</sup>D 1.4975); 17.7 g Id and 36 g POCl<sub>3</sub> in 100 ml toluene as in preceding case, 57.8% isobutylene absorbed in a solution of Br<sub>2</sub> in CCl<sub>4</sub>; CH<sub>2</sub>BrCBr(CH)<sub>3</sub>C<sub>2</sub>H<sub>5</sub>, b.p. 148-152°C, n<sup>20</sup>D 1.5112), 77.5% III and 18% tert-Card 5/7

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butyl toluene (b.p. 190-196°C, n<sup>20</sup>D 1.4940); 17.7 g Ia and 36 g POCl<sub>3</sub> in 100 ml toluene, 3 hours, 120°C, as before 2.4 g C<sub>6</sub>H<sub>5</sub>COCl (b.p. 50-80°C/14 mm; anilide, m.p. 161-162°C), 14 g of the initial substance Ia (b.p. 186-188°C/14 mm, n<sup>20</sup>D 1.5360) and 2.2 g of a substance which dissolves in concentrated H<sub>2</sub>SO<sub>4</sub>; 17.7 g Ib and 36 g POCl<sub>3</sub> in 100 ml V, 3 hours, 140°C, as before, 18% III, 6.8 g of the initial substance Ib (b.p. 165-175°C/13 mm, m.p. 56-57°C (from benzene)) and 2.3 g of a substance soluble in concentrated H<sub>2</sub>SO<sub>4</sub>; 20.5 g I and 36 g POCl<sub>3</sub> in 100 ml toluene, 3 hours, 120°C (in the bath), 13.8 g of the initial substance Ii (b.p. 200-210°C/16 mm, m.p. 44-45° (from benzene)) and 5 g of a substance soluble in concentrated H<sub>2</sub>SO<sub>4</sub>; from the reaction of Ih, k with POCl<sub>3</sub> in toluene (3 hours, 120°C) no particular substances are obtained; in an analogous reaction between 11.9 g IV and 18 g POCl<sub>3</sub> in 50 ml V (3 hours, 140°C) HCl is separated; 7.1 g of the initial substance IV are obtained, but alkylation products of V cannot be established. If POCl<sub>3</sub> is replaced

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| by 30 g P <sub>2</sub> 0                   | , 20% VI are separated   | at 120°C, 13.2% VI and 45% IV are     |
| obtained                                   | Abstracter's note: Con   | plete translation.]                   |
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AUTHORS:

Prajenar Bronisław, Troszkiewicz Czesława

TITLE:

Influence of the benzyl group structures of the amides X-C6-H5-CHR-NH-C0-C6h5 on the course of the benzylation of aromatic compounds

PERIODICAL:

Referativnyy zhurnal. Khimiya, no. 4, 1963, 241-242, abstract 42h131 (Roczn. chem., v. 36, no. 5, 1962, 843-851 Pol.; aummaries in Russ. and Eng.)

TEXT: In order to study the influence of the atructure of the ArCH(R)-group on the course of the reaction when aromatic compounds are subjected to benzylation with amides of the general formula ArCH(R)NHCOC6H5 in the presence of POCl<sub>3</sub>, toluene alkylation reactions were investigated under the influence of 2-R-3-R'-4-R"C6H2CH2NHCOC6H5 (Ia, b, c, d where (a) R=R"=H, R'=C2H5O; (b) R=C2H5O, R'=R"=H; (c) R=R'=H, R"=C2H5O; Card 1/6

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(d) R=R'=H, R"=NO<sub>2</sub>) and C<sub>6</sub>H<sub>5</sub>GH(R)NHCOC<sub>6</sub>H<sub>5</sub> (II, where R = C<sub>2</sub>H<sub>5</sub>) (IIa) as well as alkylation of benzene under the influence of II (R=C<sub>6</sub>H<sub>5</sub>) (IIb). Under the influence of POCl<sub>3</sub>, the opening of the C-N bond and the breaking off of the C<sub>2</sub>H<sub>5</sub>OC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>-group in the cases Ia, b, c take place easily with a high yield of C<sub>6</sub>H<sub>5</sub>CN (III). On the other hand, the C<sub>2</sub>H<sub>5</sub>O group decreases the yield of benzylation products of toluene, especially in case of its ortho or para position in which it is linked with the CH<sub>2</sub>-group. At the same time, it increases the relative yield of dibenzylation products for the reaction with Ia (apparently a mixture of 3-(3'-C<sub>2</sub>H<sub>5</sub>O-C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>)-4-CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub> and 2-(3'-C<sub>2</sub>H<sub>5</sub>OC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>)-5-C<sub>2</sub>H<sub>5</sub>OC<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH<sub>3</sub>-4"). The NO<sub>2</sub>-group counteracts the breaking off of the 4-NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>-group so strongly that the main reaction product obtained is the initial substance Id. The degree compared with the C<sub>2</sub>H<sub>5</sub>-group in IIa. When the latter was used,

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unsaturated hydrocarbons were found in the reaction products. 66 g 3-C<sub>2</sub>H<sub>5</sub>OC<sub>5</sub>H<sub>4</sub>CHO (IV) (b.p. 117-119°C/11 mm) are boiled in 300 ml alcohol and a colution of 85 g NH<sub>2</sub>OH·O.5 H<sub>2</sub>SO<sub>4</sub> in 300 ml of water and neutralized Na<sub>2</sub>CO<sub>3</sub> for one hour, 200 ml of the solvent are driven off, 300 ml of the added and the oxime of IV is separated (IVa), C<sub>9</sub>H<sub>11</sub>NO<sub>2</sub>, m.p. 60-61°C (from benzene). 70 g of metallic Na is as quickly as possible added to 60 g of IVa in 550 ml of absolute alcohol, the mixture is boiled for about 2 hours, 200 ml of water are added gradually, about 500 ml of the distillate are dirven off with steam and from the rest is extracted with ether giving: 90% 3-C<sub>2</sub>H<sub>5</sub>-0C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> (V), b.p. 120-122°C/13 mm, n<sup>2</sup>OD 1.5303; hydrochloride (ChHt), C<sub>9</sub>H<sub>14</sub>ClNO, m.p. 134-135°C (from dioxane); picrate (PK), m.p. 178-179°C; acetamide (AD), C<sub>11</sub>H<sub>15</sub>NO<sub>2</sub>, b.p. 197-198°C/3 mm, m.p. 60, 5-61°C (from benzene). 60 g C<sub>6</sub>H<sub>5</sub>COCl are added dropwise to the weighed quantity of 40 g V in 300 ml of 10% NaOH. This gives 69 g of Ia, C<sub>16</sub>H<sub>17</sub>NO<sub>2</sub>, m.p.

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56-57°C (from benzene-gasoline). The synthesis of I is analogous 56-57°C (from benzene-gasoline). The synthesis of the analogous (specifications are I, empirical formula, m.p. in °C): b, C16H17NO2; 100-100.5 [2-C<sub>2</sub>H<sub>5</sub>OC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, yield 76%, b.p. 119-122°C/14 mm, n<sup>2</sup>O<sub>D</sub> 1.5294; ChHt, C9H14ClNO, m.p. 164-165°, PK, m.p. 197-198°, AD, C11H15NO2, m.p. 85-86°]; b, C<sub>16</sub>H<sub>17</sub>NO<sub>2</sub>, 98-99 (4-C<sub>2</sub>H<sub>5</sub>OC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, yield 80%, m.p. 128-129°C/15 mm), and also IIb, m.p. 108-109°C [C6H5-CH(C2H5)NH2, b.p. 94-95°/19 mm]. A Schotten-Baumann reaction on 4-NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>HH<sub>2</sub> yields Ig, m.p. 155.5-156°C (from alcohol). To 0.1 moles of  $(c_6H_5)_2$ CHOH and 0.1 moles of III in 60 ml of glacial CH3COOH, 11 g of concentrated H2SO4 are added dropwise at 45°C in the course of 20 minutes, the mixture is heated to 45°C for 1 hour and then poured into 300 ml waterice mixture. This gives 91% IIa, m.p. 171-1720 (from alcohol). 22 g of Ta, b, c, 90 ml of toluene and 33.5 g of POCl3 are heated at 120°C for 3 hours; 1.69-1.74 moles of HCl are separated per mole of I; ice is added and the procedure described in the preceding paper (RZhKhim, 1960, no. 7, 26566) is carried out; the following substances Card 4/6

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separate (specifications are I, monoalkylation product, empirical formula, yield in %, b.p. in °C/mm, yield of III in %, residue in g): Ia, tolyl-benzyl)-toluene, C<sub>16</sub>H<sub>18</sub>O, 43.5, 180-182/13, 1.5625, di-(3-ethoxy-benzyl)-toluene, C<sub>25</sub>H<sub>28</sub>O<sub>2</sub>, 20.8, 265-270/12, 88.4, 3; Ib, tolyl-(2-ethoxyphenyl)-methane, C<sub>16</sub>H<sub>18</sub>O, 15, 169-172/12, 1.5595, di-(2-ethoxy-benzyl)-toluene, C<sub>25</sub>H<sub>28</sub>O<sub>2</sub>, 16.1, 220-226/1, 92, 9. Ic gave 2 g of a mixture of monoalkylation and dialkylation products, b.p. 160-240°/mm, 69% POCl<sub>3</sub> are heated at 120°C for 3 hours; after ice has been added, 17.1 g of the initial substance Id separate; distillation effects the separation of 9% III and 1.3 g of a substance having a m.p. of 170-200°C/13 mm, from which 0.5 g of 4-NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>COCl, m.p. 69-70°C, can be separated (from alcohol). 18.3 g of IIb, 80 ml of C<sub>6</sub>H<sub>6</sub> and 24 g of POCl<sub>3</sub> are heated at 120°C for 4 hours; after ice has been added, 4 g of (C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>CH (VI), m.p. 79-82°C, are separated (from alcohol); distillation gives: 76% III, b.p. Card 5/6

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 $74-76^{\circ}$ C/14 mm, 4.4 g of a mixture consisting of 13% (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>-CHCl and 87% VI (total yield of VI 50.5%) and 3-5.4 g of residue, from which 29% 1.4-[(C6H5)2CH]2C6H4, m.p. 164-165°C are separated (from glacial CH3COOH). 23.8 g of IIa, 100 ml of toluene and 37 g of POCL3 are heated at 120°C for 3 hours; in the toluene solution the presence of 16.1% of an unsaturated compound (calculated with respect to C6H5CH=CHCH3) is determined y means of 2.575 g Br<sub>2</sub>; distillation gives 80% of III; bromination with a solution of Br<sub>2</sub> in CCl<sub>4</sub> yields 1.5 g of C<sub>6</sub>H<sub>5</sub>CHBrCHBrCH<sub>3</sub>, m.p. 64-65°, (from alcohol). C<sub>16</sub>H<sub>18</sub>, yield 45.2%, b.p. 160-166°C/12 mm, n<sup>20</sup>D 1.5682 is separated from a higher fraction (9.5 g), b.p. 160-170°C/13 mm, ethylphenyln-tolyl methane. Oxidizing the higher fraction with Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in CH<sub>3</sub>COOH gives 4-C6H5COC6H4COOH, m.p. 190-192°C. [Abstracter's note: Complete

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New method of synthesis of quinoline nucleus by cyclization of oximes. Rocz ehemii 36 no.9:1387-1388 162.

1. Department of Organic Chemistry, Silesian Institute of Technology, Gliwice.

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Unification and standardization of cranes and the economic results. Przegl techn [84] no.44:5,6 4 N 162.

TROSZKIEWICZ, Z.

Technical progress in the construction of cranes. p.161

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Uncl.

